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A STUDY OF THE ROOM TEMPERATURE PHOSPHATE PROCESS



TECHNICAL REPORT

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By

Jodie Doss

June 1966

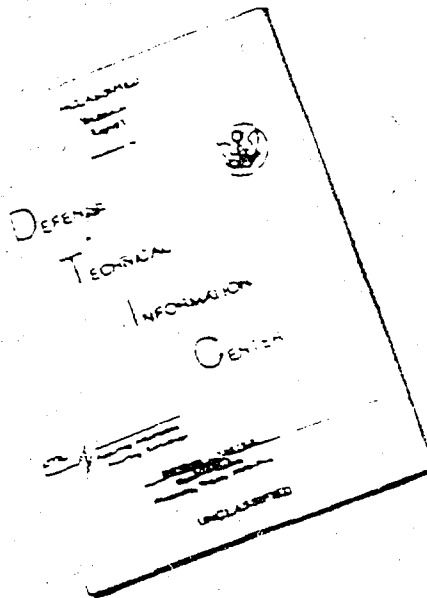
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ABSTRACT

Attempts were made to establish optimum concentration of constituents and operating conditions for the room temperature zinc phosphating process as developed at Rock Island Arsenal and to attempt to convert the conventional manganese phosphate solution into a room temperature phosphate solution.

For the room temperature zinc phosphate process the optimum pH was found to be approximately 4.

Good coatings were produced from solutions that were two to three times more concentrated in total acid than the normal operated solution.

Moderate addition of nitrite to the room temperature phosphate solution decreased the coating weight of the resultant coating but not the corrosion resistance. A large amount of nitrite decreased the ferrous iron and zinc content.

Attempts to convert a conventional manganese phosphate solution into a room temperature phosphating solution was not successful at this time.

The results of this investigation should be included in the operating procedure for the room temperature phosphate process. This information should be included in the Military Handbook 205, "Phosphatizing and Black Oxide Coatings of Ferrous Metals."

The room temperature zinc phosphate solution should be further investigated. Effort should be directed towards methods of maintaining the ferrous iron contents of the solution; improving the quality of the coating on hard steel alloy; and decreasing the amount of sludge found in the solution.

FOREWORD

During FY65 the Metal Finishing Unit, Rock Island Arsenal, was assigned a task, "Materials for Army Weapons and Combat Mobility," AMS Code 5025.11.84205, subtask "Protective and Packaging Materials." This authorized the Metal Finishing Unit to initiate and conduct a number of problems under the subtask. This report describes work under "Innovations in Phosphatizing Techniques." This problem was continued in FY 1966 under "Metals Research for Army Materiel," AMS Code 5025.11.294, "Improvements in Phosphatizing Techniques, Phase 1 Room Temperature Phosphate."

TABLE OF CONTENTS

Page

Title Page

Abstract

Foreword

Table of Contents

Problem

Background

Approach and Results

Preparation of Room Temperature Zinc
Phosphating Solution

Preparation of Test Specimens

The Effect of pH on Coating Weight

Effect of Increasing the Solution
Concentration

The Effect of Nitrite Addition to Phosphate
Solution

Effect of Nitrite Additions on Iron and Zinc
Concentration

Room Temperature Manganese Phosphate Solution

Discussion

Conclusions

Recommendations

Literature Cited

Distribution

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PROBLEM

To improve the formulation of the Rock Island Arsenal room temperature zinc phosphate solution.

To work on development of a room temperature manganese phosphate solution.

BACKGROUND

Phosphate coatings are not new. They have been available for centuries. It is reported that the Romans used phosphate coatings on iron in the third century, but they were a far cry from the modern phosphate coating. The modern coating process probably began with the Coslett British patent of 1906.⁽¹⁾

Modern phosphating is a process whereby insoluble bivalent metallic phosphates, primarily zinc and manganese, are deposited on other metallic surfaces, generally steel or iron, as a paint bonding agent or in conjunction with rust preventive compounds as a corrosion resistant coating. The coating is deposited from an aqueous solution.

The solution is usually composed of the particular bivalent metallic phosphate, an oxidizing agent, ferrous iron and phosphoric acid. The solution is controlled by analyzing for free acid, total acid, ferrous iron, oxidizing agent, and zinc or manganese metal content. The solution is normally operated between 195° and 205°F.,⁽²⁾ and is generally referred to as the conventional hot process.

The conventional hot process produces a satisfactory coating, but has several inherent disadvantages. Some of these are:

1. The cost of heating and regulation of the bath temperature.
2. The tank size limitations due to heating economics.
3. The concentration fluctuations caused by replacement of evaporated water.
4. The agitation of the bottom sludge caused by convection and by repeated stirring to mix in the replacement of evaporated water.
5. The lack of ability to set up a field tank where a suitable source of heat is not available or may be limited.

Because of these hindrances and possibly others, investigations were directed toward solutions that would coat steel at room temperature. There was a discovery of a zinc phosphate solution that would operate at room temperature but not a room temperature manganese phosphate solution.

The first patent for such a zinc phosphate solution was issued in 1941.⁽³⁾ The results of the investigation that was the basis of the patent is presented in an article by Schuster and Krause.⁽⁴⁾ They found it was possible to produce a phosphate coating at room temperature.

A later investigation by Földes⁽⁵⁾ revealed that a coating could be produced in a room temperature phosphate solution with the pH of the solution maintained between 2.6 and 2.8. Sodium fluoride was a necessary accelerator and buffer. Lapatukhin,⁽⁶⁾ utilizing the same general solution, found that the ideal solution must be maintained at a higher pH of 2.8 to 3.0.

A later investigation, utilizing a steel wool covered glass electrode submerged in a zinc phosphate solution, revealed that the ideal pH range must be between 3.4 and 4.4.⁽⁷⁾ Unreported work by the above authors narrowed the range to 3.5 to 4.0. This is the pH range by which the Rock Island Arsenal room temperature zinc phosphate solution is operated. The pH of the solution is maintained in this range by the buffer sodium fluoride (Na_2F_2). Also an additional accelerator sodium nitrite (NaNO_2) is added to the solution. This solution has been utilized for several years.

Although this range has proven adequate, the specific optimum pH was never established. This is one of the aspects of the bath that this work proposes to investigate.

There are many details about the solution that need clarification, primarily those dealing with optimum operating conditions which include optimum pH, optimum solution concentration and the effect of increasing the sodium nitrite concentration of the solution. Therefore, the present investigation was directed toward this end.

In addition, the technology of preparation and operation of the room temperature zinc phosphating solution was utilized toward trying to convert the hot manganese phosphate solution to a room temperature phosphate solution.

APPROACH AND RESULTS

Preparation of Room Temperature Zinc Phosphate Solution

This part of the study was involved in investigating significance of pH, solution concentration, and sodium nitrite on the effectiveness of the room temperature zinc phosphating solution.

A description of this basic solution is a prerequisite to discussion of the above variables.

The standard room temperature zinc phosphate solution is prepared from a nitrate accelerated hot zinc phosphate solution of the following composition range: Ferrous iron content 0.4 to 0.6 percent, nitrate content 2.0-2.5 percent.

Free Acid (FA) = 2-5 points*
(Titrating with 0.1N sodium hydroxide using modified methyl orange as indicator)

Total Acid (TA) = 24-35 points*
(Titrating with 0.1N sodium hydroxide using phenolphthalein as the indicator)

$$\text{Ratio} \quad \frac{\text{Total Acid (TA)}}{\text{Free Acid (FA)}} = 6 \text{ or greater}$$

The concentrations are adjusted routinely by adding appropriate bath ingredients.

To prepare the room temperature bath, the properly adjusted hot zinc phosphating solution is cooled to room temperature. The pH of the solution is adjusted to the desired range, normally, 3.5 to 4.0. This is accomplished by the stepwise addition of sodium fluoride (Na_2F_2) until the desired pH is obtained. The solution is now ready for the addition of the secondary accelerator, sodium nitrite (NaNO_2). Approximately 0.1 gram/liter of NaNO_2 is added to the solution, and stirred. This is the normal operating (Rock Island Arsenal) room temperature phosphating solution.

Preparation of Test Specimens

The specimens used for tests were 2" x 3" x 1/16" FS 1020 steel panels. The specimens were degreased

*A point is equivalent to one ml. of a 0.1N sodium hydroxide when titrating a 10 ml. sample.

utilizing a trichlorethylene vapor degreaser. The surface of the specimens were next steel grit blasted utilizing number 80 grit. The specimens were then placed in a desiccator until needed.

The Effect of pH On Coating Weight

The first phase of this investigation was an attempt to determine the optimum pH for the Rock Island Arsenal room temperature phosphate solution. Hot zinc phosphate solutions were secured from the regular processing tanks in the shop as needed. The solutions were analyzed and found to fall within the ranges specified in the section of this report entitled "Preparation of Room Temperature Zinc Phosphating Solution."

The pH of the phosphating solution was the first solution variable that was investigated, while the other variables such as iron, nitrite, were held as constant as possible.

Five separate samples were obtained from the shop and adjusted in 0.1 increments by the additions of sodium fluoride (Na_2F_2). At each pH value, five steel test specimens were processed for thirty minutes. The phosphated specimens were then weighed and the coating stripped by a 15 minute immersion in a 5 percent chromic acid solution, which had been heated to $165 \pm 5^\circ\text{F}$. The stripped panels were reweighed. The coating weights of the specimens were determined. The average coating weight was calculated. The results of the determinations are presented in Table . The general trend indicated that coating increased with pH to a value of pH 4. Solutions with pH values between 4.0-4.5 produced higher coating weight results. This was believed to be due to the fact that some of these values were above the pH value for normal coating format:

Effect of Increasing the Solution Concentration

The next facet of the investigation involved increasing the concentration of replenisher or concentrate in the phosphating solution to increase the total acid. Replenisher is a concentrate of zinc oxide dissolved in concentrated nitric and phosphoric acids used in the daily routine maintenance of the acidity of the phosphate solution.

For the tests, hot phosphating solutions were obtained that had analysis values within the range desired for the room temperature phosphating solution. One liter aliquots of hot solution were converted to a room temperature phosphate solution by the previously outlined procedure in this report.

TABLE I

**THE EFFECT OF pH VARIATIONS ON COATING
WEIGHT (PROCESSING TIME - 30 MINUTES)**

<u>pH</u>	<u>3.2</u>	<u>3.3</u>	<u>3.4</u>	<u>3.5</u>	<u>3.6</u>	<u>3.7</u>	<u>Solution No.</u>
Average Coating Weight (mg/ft ²)	1120	1110	1180	1320	1540	1620	1
	1140	1100	1160	1230	1390	1580	2

<u>pH</u>	<u>3.7</u>	<u>3.8</u>	<u>3.9</u>	<u>4.0</u>	<u>4.1</u>	<u>4.5</u>	<u>Solution No.</u>
Average Coating Weight (mg/ft ²)	1280	1470	1650	1600	1800	1630	3
	1510	1580	1630	1720	1690	1820	4
	1570	1550	1590	1700	1610	1630	5

under "Preparation of Room Temperature Zinc Phosphating Solution." The procedure involved adjusting the pH of the solution within the range of 3.7 - 3.9 by the addition of sodium fluoride. The pH was held to the narrower range because of the higher coating weight obtained at this range. Next was added approximately 0.1 gram NaNO₂ per liter to the solution.

Six steel specimens were processed in the one liter solution for 30 minutes. Half of the coated specimens were utilized for coating weight determinations, and the other half were tested for salt spray resistance to 5% sodium chloride in accordance with "Salt Spray Test" Method 811.1 of Federal Test Method Standard 151. The results are shown in Table II (Solution 1, Test 1).

At this point twenty milliliters of replenisher was added to the liter of solution and six more specimens were processed and evaluated as above. The information on this test is shown as Solution 1, Test 2 of Table II.

Twenty more milliliters of replenisher was added to the solution. More specimens were processed and evaluated as before. Data is shown in Table II as Solution 1, Test 3.

Data shown under Solutions 2 and 3 are from repeats of the above procedure.

TABLE II**THE EFFECT OF INCREASING SOLUTION CONCENTRATION
IN THE ROOM TEMPERATURE ZINC PHOSPHATE SOLUTION**

<u>Solution 1</u>	<u>Test 1</u>	<u>Test 2</u>	<u>Test 3</u>
% Fe ⁺⁺	0.40	0.40	0.39
Free Acid (FA)	2.3	3.5	3.2
Total Acid (TA)	24.0	30.2	34.5
Ratio $\frac{TA}{FA}$	10.4	8.6	10.8
pH	3.9	3.9	3.9
Average Coating Weight (mg/ft ²)	1280	1620	2060
Salt Spray	Failed	Passed	Passed
Results	2 hr. test	2 hr. test	2 hr. test

<u>Solution 2</u>	<u>Test 1</u>	<u>Test 2</u>	<u>Test 3</u>
% Fe ⁺⁺	0.40	0.39	0.38
Free Acid (FA)	2.7	2.6	2.4
Total Acid (TA)	29.5	34.5	39.4
Ratio $\frac{TA}{FA}$	10.9	13.3	16.4
pH	3.9	3.9	3.9
Average Coating Weight (mg/ft ²)	1490	1510	1770
Salt Spray	Passed	Passed	Passed
Results	2 hr. test	2 hr. test	2 hr. test

<u>Solution 3</u>	<u>Test 1</u>	<u>Test 2</u>	<u>Test 3</u>	<u>Test 4</u>
% Fe ⁺⁺	0.40	0.40	0.40	0.40
Free Acid (FA)	2.4	3.4	5.0	6.2
Total Acid (TA)	21.3	26.0	30.3	36.0
Ratio $\frac{TA}{FA}$	8.9	7.6	6.1	5.7
pH	3.9	3.9	3.9	3.9
Average Coating Weight (mg/ft ²)	1170	1580	1620	2070
Salt Spray	Failed	Passed	Passed	Passed
Results	2 hr. test	2 hr. test	2 hr. test	2 hr. test

Information from the three tests are also shown graphically in Figure 1.

Only the coatings from Solution 1, Test 1 with a total acid of 24 and Solution 3, Test 1 with a total acid of 21.3 failed the two hour salt spray test.

Table III shows the performance of coatings produced in solutions with total acid values between 50 and 126.3. These values were obtained by adding various amounts of replenisher. Note that satisfactory coatings were obtained over this entire range of total acid.

The Effect of Nitrite Addition to Phosphate Solution

The next aspect of this investigation was to study the effect of additions of sodium nitrite to the room temperature phosphate solution.

A hot zinc phosphate solution was converted to a room temperature phosphate solution with the exception of the addition of the sodium nitrite. This was divided into five - one liter solutions. No sodium nitrite was added to the first solution, .0125 gm of sodium nitrite was added to the second, .0625 gm to the third, 0.125 gm to the fourth and 0.625 gm was added to the fifth. Groups of four steel specimens were processed in each solution. The amount of sodium nitrite normally added has been 0.10 gram/liter.

Phosphate coating weights were determined for each group of specimens. The results are presented in Table IV. The results indicated that as the amount of nitrite is increased the resultant coating weight is decreased.

Effect of Nitrite Additions on Iron and Zinc Concentration

Other tests were conducted in order to determine the effect of nitrite additions on the iron and zinc content of the phosphate solution. Iron and zinc were determined according to "Military Handbook 205," "Phosphatizing and Black Oxide Coating of Ferrous Metals." The results are presented in Table V. The results show that as nitrite is increased there is a gradual decrease of the zinc content. A large amount of nitrite decreases the ferrous iron content.

Room Temperature Manganese Phosphate Solution

The final effort was directed toward developing a room temperature manganese phosphate solution. The conventional hot manganese phosphate solution was cooled to room temperature, analyzed and adjusted to fall within the following

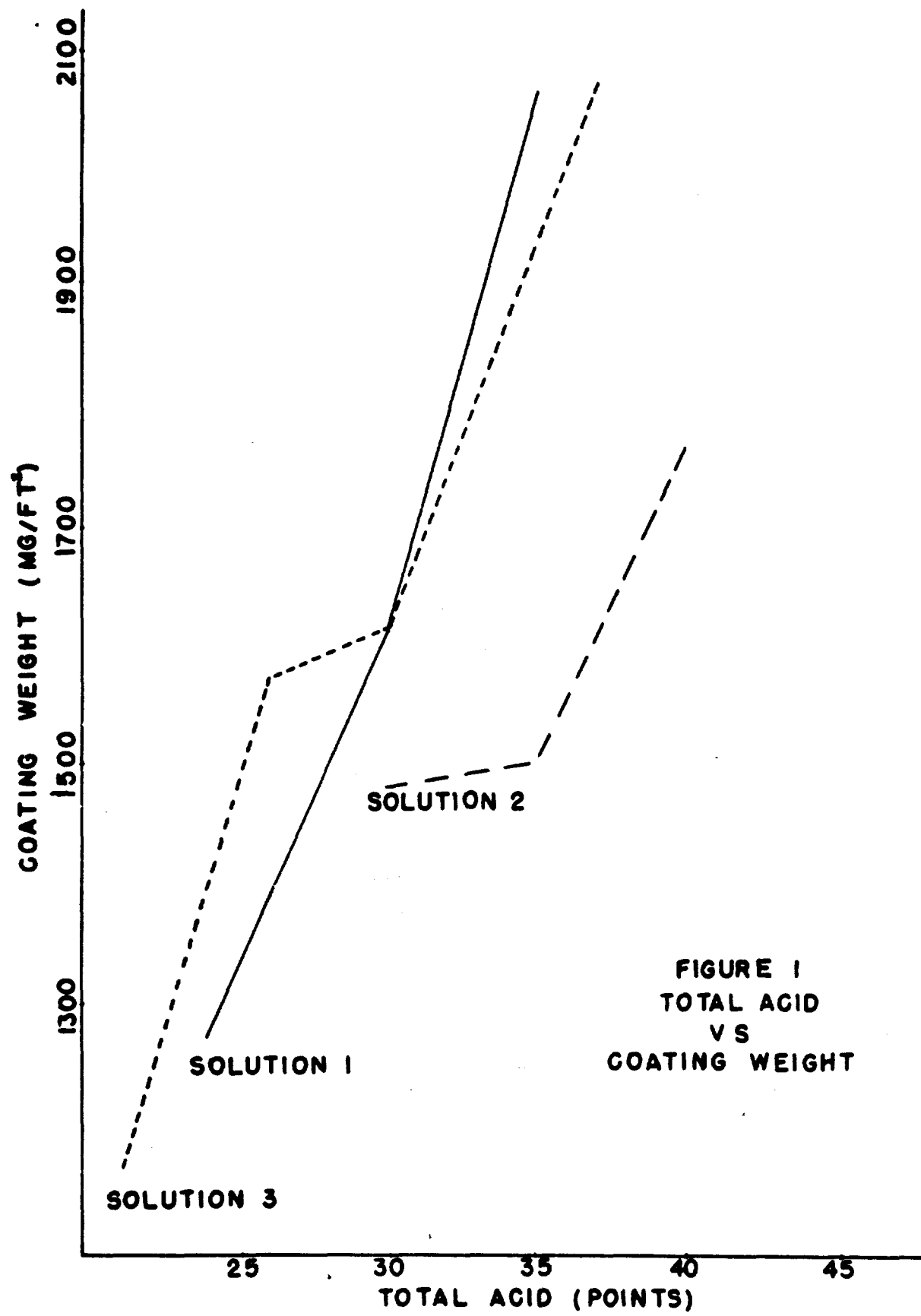


FIGURE 1
TOTAL ACID
VS
COATING WEIGHT

TABLE III

BATHS CONTAINING LARGER SOLUTION CONCENTRATIONS

[illegible]

TABLE IV

**THE EFFECT OF NITRITE ADDITION ON COATING
WEIGHT (PROCESSING TIME 30 MINUTES)**

Nitrite Addition Grams NaNO_2/liter Phosphating Solution	0	.0125	.0625	0.125	0.625	Soluti
Average						
Coating	1430	1410	1350	1250	790	1
Weight						
(mg/ft²)	1490	1410	1320	1210	680	2
	1690	1670	1580	1480	970	3

TABLE V

**THE EFFECT OF NITRITE ADDITIONS ON IRON
AND ZINC CONCENTRATION**

Nitrite Grams NaNO_2/liter Solution	Ferrous Iron gram/liter		Zinc gram/liter
	Test 1	Test 2	Test 3
0	4.4	4.1	4.87
0.0125	4.4	4.1	4.70
0.0625	4.4	4.1	4.70
0.125	4.4	4.1	4.29
0.625	4.1	3.2	4.18

ranges:

Free Acid (FA) = 2.0-5.0 points*
(Titrating with a 0.1N sodium hydroxide using modified methyl orange as the indicator)

Total Acid (TA) = 24-35 points*
(Titrating with a 0.1N sodium hydroxide using phenolphthalein as the indicator)

Ratio $\frac{TA}{FA}$ = 6 or greater

Iron (ferrous) = 0.2 to 0.3 percent

The concentrations were adjusted routinely by adding appropriate bath ingredients.

A variety of chemicals were screened by making additions to one liter aliquot solutions at room temperature to determine if the chemical would accelerate coating formation. The pH of the solution was adjusted to specific values within the range of 2.5 and 4.5 by the addition of the chemical or with an addition of a 20% sodium hydroxide solution. Steel specimens were immersed for thirty minutes in the solution at each of the different pH values. The immersed specimens were removed from the solution, inspected for coatings and subjected to the 5% salt spray test for corrosion resistance. The results of the investigation are presented in Table VI. A heavy coating was obtained from a very few of the solutions containing the added chemicals. A thin coating was obtained from a few of the solutions. All the coatings had very poor salt spray resistance.

DISCUSSION

The phosphating procedure is a complicated process. There are a number of reactions proceeding toward the ultimate end of producing the phosphate coating.⁽⁸⁾ This investigation attempts to determine some of the optimum operating conditions for the Rock Island Arsenal Room Temperature Phosphate Solution.

An early investigation⁽⁷⁾ had shown that before the phosphate coating is formed there is a rise in the pH of the solution at the interface surface of the coated metal. The initial pH of the solution is important. Therefore, the first variable that was investigated was the effect

*One point equals 1 milliliter of 0.1N NaOH per 10 milliliter sample.

TABLE VI

**RESULTS OF PROCESSING STEEL IN MANGANESE
PHOSPHATE SOLUTION CONTAINING VARIOUS ADDED CHEMICALS
(30 MINUTES PROCESS AT ROOM TEMPERATURE)**

<u>Chemicals Added</u>	<u>pH Value</u>	<u>Results</u>	<u>Salt Spray (30 min. test)</u>
		<u>Coating</u>	
Disodium Ethylene - diaminetetraacetate + CaCO ₃	2.5	No coating	Failed
	3.3	" "	"
	3.8	Very thin coating	"
Citric Acid + NaF	2.3	No coating	"
	3.4	" "	"
	3.9	Very thin coating	"
CaCO ₃ + NaF	3.0	" " "	"
	4.5	" " "	"
	4.0	No coating	"
Na ₄ P ₂ O ₇ + NH ₄ FHF	4.2	Thin coating	"
	3.0	Very thin coating	"
	3.6	" " "	"
Mn(NO ₃) ₂ + NaF + NaNO ₂	3.3	No coating	"
	3.9	" "	"
	4.2	Thin coating	"
Disodium Ethylene - diaminetetraacetate + NaNO ₂	3.0	Very thin coating	"
	3.7	" " "	"
	4.2	" " "	"
Na ₂ B ₄ O ₇	3.1	" " "	"
	3.8	" " "	"
	4.3	" " "	"
Sodium citrate	3.1	" " "	"
	3.7	" " "	"
	4.2	" " "	"
Sodium tartrate	3.0	" " "	"
	3.6	" " "	"
	4.2	" " "	"
Sodium oxalate	3.0	Heavy coating	"
	3.9	" "	"
SnCl ₂	3.1	No coating	"
	4.1	" "	"
Na ₂ SO ₃	2.8	Thin coating (H ₂ S odor)	"
	3.9	" "	"
Sodium glucoheptanate	3.0	No coating	"
	3.9	" "	"
Sn ₂ P ₂ O ₇	2.5	" "	"
	3.9	" "	"
Cobalt acetate	2.6	" "	"
	3.9	" "	"
MnO ₂	3.2	Very thin coating	"
	4.1	" " "	"

TABLE VI (Continued)

**RESULTS OF PROCESSING STEEL IN MANGANESE
PHOSPHATE SOLUTION CONTAINING VARIOUS ADDED CHEMICALS
(30 MINUTES PROCESS AT ROOM TEMPERATURE)**

<u>Chemical Added</u>	<u>pH Value</u>	<u>Results</u>	<u>Salt Spray (30 min. test)</u>
		<u>Coating</u>	
Na_2F_2	3.4	No coating	Failed
	4.1	" "	"
$\text{Al}(\text{NO}_3)_2$	3.2	" "	"
	4.0	Very thin coating	"
$\text{Mg}(\text{NO}_3)_2$	3.1	No coating	"
	4.1	" "	"
Succinic acid	3.0	Very thin black coating	"
	4.0	No coating	"
Dextrose	3.1	" "	"
	4.1	Very thin coating	"
Dextrin	3.1	No coating	"
	3.9	Thin coating	"
KClO_3	3.1	No coating	"
	3.9	" "	"
NaBO_2	3.1	" "	"
	3.7	" "	"
$\text{K}_2\text{Cr}_2\text{O}_7$	3.1	" "	"
	4.2	" "	"
Na_2SiO_3	3.0	" "	"
	4.3	" "	"
KClO_4	2.5	" "	"
	3.0	" "	"
Sodium acetate	3.2	" "	"
	3.8	" "	"
	4.0	" "	"
Sodium gluconate	3.9	" "	"
	4.1	" "	"
$\text{Cu}(\text{NO}_3)_2$	3.0	Heavy coating	"
	3.9	" "	"
I_2 + little alcohol	3.0	No coating	"
	4.0	" "	"
$\text{Na}_2\text{S}_2\text{O}_3$	3.0	No coating (H_2S odor)	"
	3.9	Thin coating	"
NaBiO_3	3.2	No coating	"
	4.2	" "	"

of pH on the coating weight. The results of this investigation revealed that as the pH for a given solution is increased there is an increase of the coating weight. The maximum coating weight appears to be deposited when the solution has a pH of approximately 4.

The addition of phosphate replenisher or concentrate to the phosphate solution, to give a more concentrated solution so far as total acid is concerned, is desired because heavy coatings were obtained from those solutions. Solutions that were two to three times as concentrated, as the normally utilized solution, gave very good results. This is considered valuable information because in order to maintain or increase the ferrous iron content of the solution it becomes necessary to continually add iron syrup ($\text{Fe} + \text{H}_3\text{PO}_4$). This adding of iron syrup raises and keeps the total acid high in the room temperature phosphate solution.

The nitrite is a secondary accelerator for the room temperature phosphate solution. Nitrite is used to increase the quality of the phosphate coating. The addition of nitrite to the solution decreased the coating weight. Yet one should be careful not to add too much nitrite for this decreases the ferrous iron and zinc content of the solution.

The next endeavor was an attempt to buffer or accelerate the manganese phosphate solution so as to coat steel at room temperature. The results of these investigations are presented in Table VI. No coating was produced that would pass one half hour salt spray test.

CONCLUSIONS

Although the phosphating process is complicated, there are certain conclusions that were evident. The pH of the solution for the maximum coating weight should be approximately 4. An increase in the solution free and total acid did not have any deleterious effect on the coating weight. The addition of nitrite to the phosphating solution decreased the coating weight of the specimen.

RECOMMENDATIONS

It is recommended that the results obtained from this investigation be included in the operating procedure for the room temperature phosphate process.

The room temperature zinc phosphate solution should be further investigated. Effort should be directed toward

methods of maintaining the ferrous iron content of the solution; improving the quality of coating on hard steel alloys, and decreasing the amount of sludge found in the solution.

The operating procedure for the Room Temperature Zinc Process should be included in the Military Handbook 205, "Phosphatizing and Black Oxide Coatings of Ferrous Metals."

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Unclassified

Security Classification

DOCUMENT CONTROL DATA - R&D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY (Corporate author)

Rock Island Arsenal
Research & Engineering Division
Rock Island, Illinois 61201

2a. REPORT SECURITY CLASSIFICATION

Unclassified

2b. GROUP

3. REPORT TITLE

A STUDY OF THE ROOM TEMPERATURE PHOSPHATE PROCESS (U)

4. DESCRIPTIVE NOTES (Type of report and inclusive dates)

5. AUTHOR(S) (Last name, first name, initial)

Doss, Jodie

6. REPORT DATE

June 1966

7a. TOTAL NO. OF PAGES

29

7b. NO. OF REFS

8

8a. CONTRACT OR GRANT NO.

a. PROJECT NO.

DA No. 1C024401A328

c.

AMS Code No. 5025.11.294

d.

8b. ORIGINATOR'S REPORT NUMBER(S)

RIA 66-1858

8c. OTHER REPORT NO(S) (Any other numbers that may be assigned to this report)

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13. ABSTRACT Attempts were made to establish optimum concentration of constituents and operating conditions for the room temperature zinc phosphating process as developed at Rock Island Arsenal and to attempt to convert the conventional manganese phosphate solution into a room temperature phosphate solution. For the room temperature zinc phosphate process the optimum pH was found to be approximately 4. Good coatings were produced from solutions that were two to three times more concentrated in total acid than the normal operated solution. Moderate addition of nitrite to the room temperature phosphate solution decreased the coating weight of the resultant coating but not the corrosion resistance. A large amount of nitrite decreased the ferrous iron and zinc content. Attempts to convert a conventional manganese phosphate solution into a room temperature phosphating solution was not successful at this time. The results of this investigation should be included in the operating procedure for the room temperature phosphate process. This information should be included in the Military Handbook 205, "Phosphatizing and Black Oxide Coatings of Ferrous Metals." The room temperature zinc phosphate solution should be further investigated. Effort should be directed towards methods of maintaining the ferrous iron contents of the solution; improving the quality of the coating on hard steel alloy; and decreasing the amount of sludge found in the solution. (U) (Author)

DD FORM 1473
1 JAN 64

Unclassified

Security Classification

14 KEY WORDS	LINK A		LINK B		LINK
	ROLE	WT	ROLE	WT	ROLE
Phosphatizing Coating for Steel Coatings Protective Treatments Conversion Coatings					

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